(19) World Intellectual Property Organization International Bureau





(43) International Publication Date 6 March 2003 (06.03.2003)

PCT

(10) International Publication Number WO 03/018236 A1

(51) International Patent Classification7:

(

B22D 17/20

- (21) International Application Number: PCT/AU02/01140
- (22) International Filing Date: 23 August 2002 (23.08.2002)
- (25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data: PR 7214 29305/02

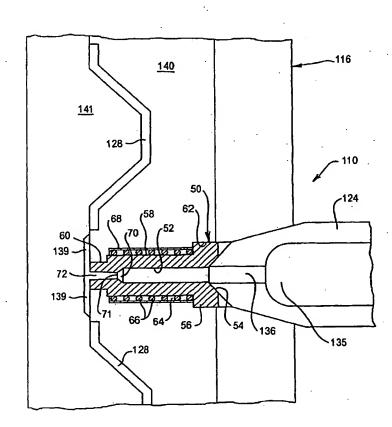
23 August 2001 (23.08.2001) AU 28 March 2002 (28.03.2002) AU

(71) Applicant (for all designated States except US): COM-MONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION [AU/AU]; Limestone Avenue, Campbell, Australian Capital Territory 2612 (72) Inventors; and

- (75) Inventors/Applicants (for US only): ROHAN, Patrick, William [AU/AU]; Unit 5, 197 Little Malop Street, Geelong, Victoria 3220 (AU). MURRAY, Morris, Taylor [AU/AU]; 51 Teal Lane, Briar Hill, Victoria 3088 (AU). COPE, Matthew, Alan [GB/AU]; 118 Jones Road, Somerville, Victoria 3912 (AU).
- (74) Agent: PHILLIPS ORMONDE & FITZPATRICK; 367 Collins Street, Melbourne, Victoria 3000 (AU).
- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

[Continued on next page]

(54) Title: IMPROVED MAGNESIUM ALLOY CASTINGS



(57) Abstract: A magnesium alloy pressure casting, produced from a magnesium alloy able to form a dendritic primary phase, has a microstructure characterised by fine primary particles in a matrix of secondary phase. The primary particles are of a form from the group consisting of rounded, spheroidal, degenerate dendritic and mixtures thereof, and are evenly distributed, while the primary particles may be substantially less than 40 μm in size, such as about 10 μm or less. The microstructure is able to exhibit alloy element separation for elements differing sufficiently in density.

WO 03/018236 A1



(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

10

15

20

25

30

IMPROVED MAGNESIUM ALLOY CASTINGS

This invention relates to improved high pressure castings of magnesium alloys.

In our Australian provisional patent application PR7218, entitled "Metal Flow System", filed on 23 August 2001 and its associated Australian complete application AU-29307/02 filed 28 March 2002, and in an International (PCT) patent application (attorney reference IRN 675213) simultaneously with the present application, there is disclosed a metal flow system for use in preparing high pressure castings from a range of suitable alloys, including magnesium alloys. The flow system of that co-pending application is for use in pressure casting of an alloy, using a pressure casting machine. The system includes a mould or die tool component which defines at least part of a flow path along which alloy, received from a pressure source of the machine, is able to flow for injection into at least one die cavity defined by a mould or die. The mould or die tool component defines, as part of the flow path, a runner which communicates with an inlet end to a controlled expansion port (a CEP) which increases in crosssectional area from the inlet end to an outlet end of the CEP. With use of the CEP the state of alloy in its flow therethrough is able to be modified from a molten state to a semi-solid state possessing thixotropic properties and that semi-solid state is able to be maintained into the or each die cavity. The CEP has a form such that, with sufficiently rapid solidification of alloy in the die cavity and back along the flow path into the CEP, alloy solidified in the CEP has a microstructure which, in longitudinal sections, is characterised by striations or bands extending transversely with respect to the alloy flow therethrough, with the bands resulting from alloy element separation, and with alternate bands relatively richer in respective elements and in primary and secondary phases respectively.

The specific form of the CEP required in the metal flow system of the provisional application PR7218, its associated complete application AU-29307/02 and its corresponding PCT application (IRN 675213) results in metal solidified in the CEP having a unique microstructure. However a casting which is solidified sufficiently rapidly to achieve this also is able to have a unique microstructure having some similarity to the microstructure of the CEP. It is the microstructure obtainable in such castings, specifically those of magnesium alloys, to which the present invention is directed. While the metal flow system is applicable to a range

15

20

25

30

of suitable alloys, such as aluminium, zinc, copper and magnesium alloys, there is a difference in its use for magnesium alloys compared with the others. Accordingly, other suitable alloys are the subject of a further provisional application PR7217 filed on 23 August 2001 and entitled "Improved Alloy Castings" and also the subject of its associated Australian complete application (attorney reference IRN 665757) filed simultaneously with the present application.

The present invention, in broad terms, provides a magnesium pressure casting of a magnesium alloy able to form a dendritic primary phase, wherein the casting has a microstructure characterised by fine primary particles in a matrix of secondary phase. The primary particles have a form from the group consisting of rounded, spheroidal, degenerate dendritic and mixtures thereof and preferably are less than 40 μm in size. Most preferably the primary particles are substantially less than 40 μm in size, such as about 10 μm or less. The primary particles are small, while they also are evenly distributed substantially throughout the casting.

The microstructure required by the invention necessitates that the casting is produced by the flow of alloy, into a die cavity in which the casting is produced, in a semi-solid state possessing thixotropic properties and does not necessitate special alloys or casting machines. This is able to be achieved by the use of a metal flow system as disclosed in the above-mentioned Australian provisional application PR7218, its associated complete application AU-29307/02 and its PCT application (IRN 675213). The microstructure also necessitates solidification of the alloy in the die cavity under conditions which both preclude significant heating of the alloy in the die cavity and relatively rapid solidification. For each of these requirements, the conditions are to be such that normal dendritic growth of primary particles in alloy flowing into the die cavity is substantially precluded.

The microstructure of a casting according to the invention differs from that obtainable in castings produced by conventional pressure die casting flow systems. In that conventional process, alloy flows into a die cavity in a substantially molten state, and primary particles present in the microstructure of a resultant casting form by normal dendritic growth. Thus, while the rate at which the alloy is cooled in the die cavity will influence the size of those particles, they still will have a conventional, normal dendritic form. As indicated, the primary particles of a casting according to the invention are of degenerate dendritic, rounded and/or spheroidal form.

15

20

25

30

The microstructure of a casting according to the invention most preferably exhibits alloy element separation for elements differing sufficiently in density, and this also is achieved by use of the metal flow system disclosed in the abovementioned provisional PR7218, its associated complete application AU-29307/02 and its PCT application (IRN 675213). This alloy element separation is a feature which, when present, further distinguishes the microstructure from one obtained by conventional pressure die casting. In that conventional process, there of course is alloy element separation in accordance with the extent to which each alloy element is present in different phases formed under given solidification conditions. However this is not on the basis of different densities *per se*, and the separation of elements differing sufficiently in density exhibited by the microstructure of a casting according to the invention is a feature additional to the separation based on phases formed. This is illustrated in the following by reference to a magnesium alloy having aluminium as a principal alloy element.

With conventional die casting of a Mg-Al alloy, for example, a resultant casting has a dendritic primary phase in a secondary phase, with the secondary phase possibly including intermetallic particles. With such microstructure, there will be a higher magnesium content and a lower aluminium content in the primary phase relative to the secondary phase. Also, the primary phase particles will exhibit a progressively increasing ratio of aluminium to magnesium content in In contrast to this, the directions extending outwardly from their centres. microstructure of a casting according to the invention most preferably will exhibit a greater separation of magnesium and aluminium between the primary phase and the secondary phase, while the primary phase will exhibit a fluctuating variation in the ratio of aluminium to magnesium content in directions extending outwardly from their centres. This separation is on the basis of the difference in density between magnesium and aluminium, and results from conditions generated in the CEP and a carry-over of those conditions in alloy flowing into and solidifying in the die cavity. A similar separation will occur between magnesium and other alloy elements of greater density. Conversely, less dense alloy elements, to the extent that the limited range is used in magnesium alloys, will tend to separate with magnesium from more dense alloy elements. The separation thus applies in general, with magnesium and any other less dense alloy element concentrating more in the primary phase and the more dense alloy elements concentrating more

10

15

20

25

30

in the secondary phase or phases, including any intermetallic secondary phase. Thus, while the primary phase shows a fluctuating variation in concentration ratio of less and more dense elements, there also is a tendency for the primary phase overall to have a lower content of more dense elements, such as aluminium.

The fluctuating variation in alloy elements from the core or centre of the degenerate dendrite particles can be more of a decaying sinusoidal form, instead of being gradual and substantially uniform as obtained in the normal dendritic primary phase obtained with conventional die casting. Thus, while the core or centre of the primary particles is richer in the less dense elements, in particular the primary element magnesium, and relatively low in the more dense elements, such as aluminium, the more dense elements may first rise, then fall and thereafter can rise again in directions outwardly from the core or centre. Thus, with a magnesium alloy such as AZ91D, the particles are low in aluminium at the core or centre but, from there, the aluminium content is able to initially increase relative to magnesium over about an initial third of the radius of the degenerate dendrite particles, then decrease relative to magnesium over about the second third of the radius, and thereafter increase again to the outer perimeter of the particles.

The fluctuating ratio of primary and secondary alloy elements in the degenerate dendrite primary particles of the casting microstructure of the invention results from the conditions able to be generated by the CEP. This is found in practice. However, it also is believed to be supported by computer simulations of flow conditions through a CEP. The computer simulations indicate . that, with flow of alloy through a suitable form of CEP which achieves relatively high flow rates through the CEP, intense pressure waves are generated in the alloy. The simulations indicate that the pressure waves are at a level of about +400 MPa. It is known that pressure differences of the order of a few 100 kPa can cause separation of less and more dense elements of an alloy, such as The computer simulations therefore point to magnesium and aluminium. pronounced separation, with movement of a less dense element to high pressure pulses and of a higher density element to low pressure pulses. Moreover, the computer simulations suggest that the intense pressure waves will have a wavelength of the order of 40 µm and will result in a microstructure exhibiting striations or bands, in longitudinal sections, which extend transversely with

10

15

20

25

30

respect to the metal flow direction. This is found to accord very closely with results able to be achieved in practice. Thus, it is found in practice that, for alloy solidified in the die cavity, under conditions providing for relatively rapid solidification in the die cavity and back into the CEP, the alloy solidified in the CEP has a microstructure in longitudinal sections which exhibits resultant striations or bands which extend transversely of the CEP. The striations or bands are found to have a wavelength of about 40 μm . That is, the spacing between centres for successive like bands, of primary element or secondary element, is of the order of 40 μm .

Thus according to the present invention, there also is provided a process for producing a casting according to the invention, using a pressure casting machine having supply means for providing in a molten state magnesium alloy which is able to form a dendritic primary phase, a mould defining a die cavity of a shape required for the casting, and a flow path providing communication between the supply means the die cavity, with part of the length of the flow path defining a controlled expansion port (hereinafter a "CEP") which increases in cross-sectional area in the direction of alloy flow along the flow path to the die cavity; wherein the process includes the steps of:

- (a) causing molten alloy to flow from the supply means into the flow path such that, in its flow from the inlet end to the outlet end of the CEP, the alloy decreases in flow velocity whereby it is caused to undergo a change in state from the molten state to a semi-solid state;
- (b) maintaining the alloy in the semi-solid state substantially throughout its flow into the die cavity; and
- (c) causing solidification of alloy in the die cavity, and back along the flow path towards or into the CEP, at a sufficiently rapid solidification rate whereby the resultant casting has a microstructure characterised by fine primary particles in a matrix of secondary phase, with the primary particles of a form from the group consisting of rounded, spheroidal, degenerate dendritic and mixtures thereof, and substantially evenly distributed.

As indicated above, a suitable CEP has an inlet by which it receives a flow of molten alloy from a runner, while it increases in cross-sectional area along its length to its outlet, in the direction in which alloy flows therethrough to fill a die cavity. In order to achieve modification of the alloy so that the alloy achieves a

15

20

25

semi-solid state possessing thixotropic properties, the inlet needs to be of a size providing for an alloy flow velocity therethrough which is in excess of about 60 m/s, such as from 140 to 165 m/s. Also, the CEP is to achieve a reduction in alloy flow velocity as the alloy flows through the CEP such that the flow velocity at the outlet is from about 50 to 80%, such as from 65 to 75%, of the flow rate through the inlet. Thus, the flow rate may be in excess of about 30 m/s through the outlet, but preferably is from about 70 to about 130 m/s, such as from about 90 to 125 m/s.

A CEP giving rise to a required change in state of the alloy may be relatively short. Depending on the size of a casting to be made, the CEP length may, for example, be from about 5 mm to about 40 mm in length, such as from 5 to 20 mm and preferably about 10 mm to 15 mm in length. The length of the CEP may define an inlet to the die cavity. Alternatively, in addition to the runner communicating with the CEP inlet, (herein referred to as the first runner), there may be a second runner providing communication between the CEP outlet and the die cavity. In another alternative, possible if a region of the die cavity is of a suitable form, that region may define at least a part of the length of the CEP. In general, it is convenient for the CEP to be circular in transverse cross-section, with the CEP being of frusto-conical form overall. However, particularly where a region of a die cavity defines at least part of its length, the CEP can be of other cross-sectional forms, such as rectangular. The suitable length and size of a CEP can vary with the selected alloy.

The metal flow system may have a single CEP from which two or more second runners extend. The second runners may communicate with a common die cavity, or with a respective die cavity. Also a single, or respective first runner may communicate with two or more CEPs of the metal flow system, with each CEP communicating directly or via a respective second runner with a common or a respective die cavity.

A pressure casting machine with which the metal flow system is used, in producing a casting according to the present invention can be of a number of different types. It may be a conventional cold- or hot- chamber high pressure die casting machine or variations thereof. Alternatively, the machine may be of the type disclosed in our Australian provisional patent application PR7216 entitled "Apparatus for Pressure Casting" and filed on 23 August 2001, its associated

10

15

20

25

30

Australian complete application AU-29303/02 filed 28 March 2002 and in an International (PCT) application (attorney reference IRN 675225) filed simultaneously with the present application.

With each suitable type of machine, magnesium molten alloy is supplied under pressure by the machine, for flow of the alloy along the flow path defined by the metal flow system and into the or each die cavity of mould or die. In general, such supply is by means of a nozzle which is secured in communication with the mould or die tool component defining the metal flow system. That component may be a mould or die half, preferably the fixed mould or die half, of the mould or die. Alternatively, the component may be an insert, such as in the form of a nozzle extension, which is engaged in such mould or die half.

Particularly where a cold-chamber die casting machine is used, larger primary particles can form in the shot sleeve and these can be carried through into a casting. Generally the volume fraction of such larger primary particles is low, although the particles can range in size from 60 μ m up to 100 μ m.

In the metal flow system used in providing a casting according to the present invention, the first runner and other parts, including the machine nozzle, of the overall metal flow system upstream from the CEP inlet, with respect to the direction of alloy flow, preferably have cross-sectional areas which are not less than the cross-sectional area of the CEP inlet. Most conveniently, those other parts of the overall flow system have cross-sectional areas which are larger than that of the CEP inlet. Preferably, a second runner, where provided, has a cross-sectional area which is not less, and most conveniently is larger, than the cross-sectional area of the CEP outlet.

Reference now is directed to the accompanying drawings in which:

Figure 1 illustrates one form of metal flow system suitable for use in producing castings according to the present invention;

Figure 2 is similar to Figure 1, but illustrates an alternative form of metal flow system;

Figure 3 is a photomicrograph showing representative microstructure of a magnesium alloy casting according to the present invention; and

Figure 4 is a photomicrograph showing the microstructure of magnesium alloy solidified in a CEP in producing a casting having a microstructure of the form illustrated in Figure 3.

15

20

25

Figure 1 illustrates one arrangement for modification of a conventional pressure casting machine, identified generally at 110. In the detail shown, it can be seen that machine 110 has a supply nozzle 124 which defines the outlet end of molten magnesium alloy supply means (otherwise not shown). From nozzle 126, the alloy is injected into die cavities 128 defined by the tools 140, 141 of mould 116.

The modification of Figure 1 involves the provision of an electrically heatable nozzle or extension 50 mounted between the outlet end of bore 136 of nozzle 124 and a respective secondary runner 139 for each die cavity 128. The nozzle 50 defines a bore 52 which provides a continuation of bore 136 and communicates with each secondary runner 139.

The nozzle 50 defines a frusto-conical seat 54 which leads to the inlet end of its bore 52. The outlet end of nozzle 124 has a complementary frusto-conical external surface which provides a seal against seat 54. The external surface of nozzle 50 is stepped to define a peripheral flange 56 around the inlet end of bore 52, an intermediate portion 58 which extends from flange 56 over a major part of the length of bore 52 and a small diameter terminal end portion 60 around the outlet end of bore 52. The fixed die tool 140 of mould 116 defines a somewhat similarly stepped recess 62 in which nozzle 50 is mounted, with flange 56 and end portion 60 being a firm friction fit in recess 62. However, intermediate portion 58 of nozzle 50 is of lesser diameter than the corresponding part of recess 62, so as to define an insulating annular air-gap 64 therebetween. Around intermediate portion 58 of nozzle 50, an electrical induction or resistance heating coil 66 is provided in air-gap 64 to enable controlled heating of nozzle 50, while a sheath of insulation 68 is provided around coil 66, against the wall of recess 62, to minimise loss of heat energy to die tool 140.

The arrangement shown in Figure 1 shows bore 52 of nozzle 50 as having a reduced size at its outlet end. This is highly desirable but, in an alternative arrangement, bore 52 may be of constant form throughout its full length. In the arrangement shown, bore 52 is of constant cross-section along a major part of its length, over which it provides a continuation of the cross-section of bore 136 of nozzle 124. However, beyond that major part of its length, in the direction of alloy flow therethrough from nozzle 124, bore 52 has a part 70 which tapers frusto-conically to a minimum cross-section at a constriction 71, and thereafter has a

part 72 which tapers frusto-conically to a cross-section at the outlet end which is larger than that at constriction 71. The part 72 comprises a controlled expansion port (or CEP) as detailed herein, while the constriction 71 is to define the location of an interface between magnesium alloy which has solidified on completion of a casting operation and alloy which is still partly in a liquid state. That is, constriction 71 establishes the interface back to which alloy solidifies to give rise to metal which separates with a casting. The inlet to the CEP is at constriction 71, while the outlet of the CEP is at the other, larger cross-sectional end of frusto-conical part 72.

As indicated the arrangement of Figure 1 comprises part of machine 110 which, apart from the differences described with reference to Figure 1, is similar to a conventional pressure casting machine. In use of machine 110, a supply of molten magnesium alloy is held in a suitable source from which the molten alloy is caused to flow through nozzle 124 and into bore 52 of nozzle 50.

10

15

20

25

30

The molten magnesium alloy, when passing through the CEP, undergoes a substantial change in its flow regime. The increasing cross-section of area of the CEP between the inlet and outlet causes a reduction in alloy flow velocity. The effective cross-sectional area of the CEP at its outlet end preferably is from two to four times greater than the cross-sectional area of the inlet end of the CEP or immediately upstream of the CEP relative to the alloy flow direction. The magnesium alloy flow velocity through the inlet end of a CEP is relatively high, such as in excess of 60 m/s, preferably from 140 to 165 m/s; with the outlet end flow velocity from about 50% to 80%, such as from 65% to 75%, of the inlet end flow velocity. Also, the flow path between the inlet and outlet of a CEP is relatively short, for example from 5 to 40 mm, such as from 5 to 20 mm, and preferably from 10 to 15 mm, such that the residence time for alloy in a CEP is very short, such as from about 60 to 100 µs for magnesium alloy flowing through the outlet end of a CEP at a preferred flow velocity.

With the rapid reduction in flow velocity in the CEP, the magnesium alloy undergoes a change in its state, from a molten state to a semi-solid state. The reduction in flow velocity also is found to be capable of generating high pressure waves in the magnesium alloy. The form of the CEP, for a given alloy, most preferably is chosen to ensure that such pressure waves are generated. Computer simulations of flow through a CEP have indicated that pressure waves

15

20

25

30

of about \pm 400 MPa can be generated. It is known that pressure differences of the order of a few 100kPa can cause separation of less and more dense elements of a magnesium alloy, such as of magnesium and aluminium. The computer simulations therefore point to pronounced separation, with migration of a less dense element to high pressure pulses and of a higher density element to low pressure pulses. Moreover, the computer simulations suggest that the pressure waves will have a wavelength of about 40 μ m.

The results of the computer simulation are found to be supported by examination of microstructures achieved with use of a suitable CEP. On completion of a casting operation, relatively rapid solidification of the alloy in and back from each die 128 cavity, is able to continue along each runner 139 and through the CEP of bore part 72 to a solid-liquid interface at or just short of the constriction at 71. With such solidification, the microstructure of alloy solidified in the CEP is found to exhibit transverse striations or bands resulting from alloy element separation. The microstructure is found to have successive bands richer in respective elements of the alloy, due to segregation on the basis of density, indicating generation of intense pressure waves in alloy in its flow through the CEP. The bands are found to have a wavelength of the order of 40 µm, indicative of pressure waves of about ± 400 MPa. Moreover, the banding to a substantial degree can involve segregation of primary and secondary phases. Thus, in the case of a magnesium alloy containing aluminium as a principal alloy element, there can be obtained alternate magnesium-rich and aluminium-rich bands, with these respectively being dendrite rich and secondary phase rich. Within aluminium-rich bands, there can be an excess of secondary phase intermetallics such as Mg₁₇ Al₁₂. Moreover, the magnesium-rich bands are found to contain primary phase as rounded, spheroidal and/or degenerate dendrite particles substantially smaller in size than 40 μm, such as about 10 μm.

The striations or bands generally extend across the full lateral extent of the CEP, substantially at right angles to the alloy flow direction. Also, they generally are evident along the full length of the CEP in that direction.

The cross-sectional area of a CEP at its inlet end, and of the metal flow path upstream from that end, preferably is small in relation to the cross-sectional area of a runner used in producing a casting of a given size by a conventional die casting process. Thus, in use of the present invention, it is preferred that the

15

20

25

30

nozzle 124 of the machine 110 be modified to a form having a bore of smaller cross-section than that used in conventional pressure die casting.

The heating coil 66 can assist in maintaining the alloy in a molten state up to the stage of its flow through the CEP. However, coil 66 has a further function. The semi-solid state of the magnesium alloy in which it has thixotropic properties, attained in its flow through the CEP, is able to be retained by the alloy during the filling of each die cavity 128. For optimum properties in the casting produced in each cavity 128, the mould 116 preferably provides for relatively rapid solidification of alloy in each cavity, such that substantially throughout each casting it has a microstructure having fine rounded, spheroidal and/or degenerate dendrite primary particles in a secondary phase or matrix. To assist in achieving this, the solidification with such microstructure preferably progresses back to the CEP to achieve a somewhat similar microstructure in alloy solidified in the CEP. However, the microstructure obtained in a CEP of preferred form also is able to be characterised by transverse striations or bands as detailed above. In both the casting and the CEP, the primary particles preferably are substantially less than 40 μm, such as about 10 μm or less.

Solidification of alloy back into the CEP is assisted by terminal end portion 60 of nozzle 50 being a friction fit in recess 52 of die tool 140, such that there is good thermal conduction from portion 160 to die tool 140. Thus, with die tools 140, 141 of mould 116 being such as to provide for rapid solidification of alloy in each die cavity 128, die tool 140 needs to be at a relatively low temperature such that it extracts heat energy from end portion 60. This assists in solidification of alloy in the CEP. However, intermediate portion 58 of nozzle 50 is insulated from die tool 140, other than for a degree of heat energy loss through portion 60, by provision of air-gap 64 and insulation sheath 68. It is in this context that heating coil 66 serves a further function. Coil 66 principally provides heat energy to intermediate portion 58 of nozzle 50. It is used to ensure that alloy in bore 52, upstream from the constriction 71, is maintained at a temperature to enable solidification of alloy in the CEP to progress back to a solid-liquid interface at or slightly downstream of constriction 71, as detailed above. Thus, liquid alloy is able to be retracted from that interface sufficiently to enable the solidified metal to be removed with the castings, upon opening of mould 116.

10

. 15

20

25

30

Turning now to Figure 2, the arrangement shown therein is similar in many respects to that of Figure 1. Thus, corresponding parts have the same reference numeral, plus 100.

In the detail shown it can be seen that the machine 210 of Figure 2 has a nozzle 224 through which the molten magnesium alloy is advanced from a source of supply (not shown). From nozzle 226, the alloy is injected into a die cavity 228 defined by the tools 240, 241 of mould 216.

The arrangement of Figure 2 has a nozzle or extension 150 mounted between the outlet end of bore 236 of nozzle 224 and a runner 239 for the die cavity 228. The nozzle 150 defines a bore 152 which provides a continuation of bore 236 and communicates with the runner 239.

The nozzle 150 defines an externally frusto-conical inlet portion 80 which defines the inlet end of its bore 152. The outlet end of nozzle 224 defines a complementary frusto-conical recess 82 which provides a seat in which portion 80 provides a seal. The external surface of nozzle 150 is stepped to define a peripheral flange 84 which is beyond the inlet portion 80 and which is a friction fit in recess 85 defined by fixed platen 86, beyond nozzle 224. Also, the outlet end of nozzle 150 is tapered and provides a seal in a complementary recess 89 defined by fixed die tool 240 of mould 216.

The arrangement shown in Figure 2 shows bore 152 of nozzle 150 as having a reduced size at its outlet end. This is highly desirable but, in an alternative arrangement, bore 152 may be of constant form throughout its full length. In the arrangement shown, bore 152 is of constant cross-section along a part of its length, over which it provides a continuation of the cross-section of bore 236 of nozzle 224. However, beyond that part of its length, in the direction of alloy flow therethrough from nozzle 224, bore 152 has a part 170 which tapers frusto-conically to a minimum cross-section at constriction 171, and thereafter has a part 172 which tapers frusto-conically to cross-section at the outlet end which is larger than that at constriction 171. The part 172 comprises a controlled expansion port (or CEP) as detailed herein, while the constriction 171 is to define the location of an interface between alloy which has solidified on completion of casting operation and alloy which is still molten. That is, constriction 171 establishes the interface back to which alloy solidifies to give rise to metal which separates with a casting.

10

15

20

25

30

Operation with apparatus 20 of Figure 2 is similar to that described with reference to apparatus 110 of Figure 1. A principal difference is that nozzle 150 of apparatus 210 is not provided with a separate heating coil, while it is in good surface to surface contact at respective parts with each of nozzle 236, fixed platen 86 and die tool 240. The arrangement is such that a sufficient thermal gradient is able to be established between the inlet and outlet ends of nozzle 150 to achieve a solid-liquid interface at or adjacent to constriction 171 when, on completion of a casting cycle, alloy solidifies in the die cavity 128 and back into the CEP defined in part 172 of the bore 152 of nozzle 150. This is able to be achieved by heat energy provided at the inlet end from nozzle 226, and heat energy extracted at the outlet end by die tool 240. It also may be necessary for heat energy to be extracted via flange 84 by fixed platen 86, and for an insulating sheath to be provided between nozzle 226 and platen 86.

Nozzle 150 of the arrangement of Figure 2 may be made of a suitable metal or ceramic. However nozzle 50 of Figure 1 preferably is made of a suitable metal.

It is indicated above that nozzle 50 of Figure 1 may have a bore 52 of constant cross-section throughout, while the same is indicated for bore 152 of nozzle 150 of Figure 2. However, in each case, this requires that a suitable CEP is provided in the alloy flow path downstream of the respective nozzle.

Each of Figures 1 and 2 show use of the metal flow system in relation to a respective of machine 110, 210, and it is to be appreciated that the type of machine can vary. Thus, in alternatives, each of the nozzles 124 and 224 is to be understood as representing the nozzle of a hot-chamber high pressure die casting machine, or the shot-sleeve of a cold-chamber high pressure die casting machine. Alternatively, either nozzle is to be understood as representing the output nozzle or conduit of a machine as disclosed in our Australian provisional application PR7216, its associated Australian complete application AU-29303/02 and its PCT application (attorney's reference IRN 675225) and the disclosures of those applications are hereby incorporated herein by reference.

Figure 3 is a photomicrograph illustrating a typical microstructure of a casting produced with use of the present invention, from AZ91 magnesium alloy. This microstructure shows fine, rounded, spheroidal and/or degenerate dendrite primary particles substantially less than 10 μ m in size and occupying up to 60% of

10

15

20

25

the volume fraction. At least most primary particles contain concentration rings showing a fluctuating, sometimes somewhat decaying sinusoidal ratio of constituent alloy elements. Between the primary particles, there is solidified metal of eutectic composition, with the fineness of the eutectic structure difficult to resolve despite the level of magnification used.

Figure 4 is a photomicrograph of the microstructure of AZ91 magnesium alloy solidified in a CEP, in producing a casting such as illustrated in Figure 3. The direction of alloy flow through the CEP is shown by an arrow. photomicrograph shows banding or striations extending transversely with respect to the flow direction. While not very readily discernible in this instance, the bands or striations as shown by X-ray analysis using secondary electron microscopy result from segregation of the parent metal magnesium and alloy additive elements such as aluminium. This segregation occurs due to intense pressure waves generated in the CEP by the reduction in alloy flow velocity as it flows through the CEP. The dynamic environment provided by the pressure waves is believed to lead to nucleation of primary particles of the parent metal at relatively high temperatures. Alternate bands are found to have a higher percentage of parent metal and a higher solidification temperature than would be expected for the starting alloy, relative to primary particles obtained in sprue/runner metal obtained by conventional pressure die casting. Similarly, the secondary phase rich intervening bands have a higher percentage of alloy elements and a lower solidification temperature than expected for the alloy, relative to secondary phases obtained in sprue/runner metal of conventional die casting. microstructure is characterised by fine primary particles substantially smaller than 10 μm in a secondary phase matrix, with a banding wavelength of about 40 $\mu m.$

Finally, it is to be understood that various alterations, modifications and/or additions may be introduced into the constructions and arrangements of parts previously described without departing from the spirit or ambit of the invention.

20

CLAIMS:

- 1. A magnesium alloy pressure casting, produced from a magnesium alloy able to form a dendritic primary phase, wherein the casting has a microstructure characterised by fine primary particles in a matrix of secondary phase, and wherein the primary particles are of a form from the group consisting of rounded, spheroidal, degenerate dendritic and mixtures thereof, and are evenly distributed.
- 2. The casting of claim 1, wherein the primary particles are less than 40 μm in size.
 - 3. The casting of claim 1, wherein the primary particles are substantially less than 40 μm in size.
- 15 4. The casting of claim 1, wherein the primary particles are about 10 μm or less.
 - 5. The casting of any one of claims 1 to 4, wherein the primary particles are distributed substantially throughout the casting.
 - 6. The casting of any one of claims 1 to 5, wherein the microstructure exhibits alloy element separation for elements differing sufficiently in density.
- 7. The casting of any one of claims 1 to 5, wherein the microstructure exhibits
 25 alloy element separation which differs from that attributable to the phases present
 and which is attributable to difference in alloy element densities.
 - 8. The casting of claim 6 or claim 7, wherein the microstructure exhibits enrichment of the primary particles in magnesium and any alloy elements less dense than magnesium and enrichment of the secondary phase in elements more dense than magnesium, relative to alloy element contents for the primary and secondary phases obtainable with a conventional casting of the same magnesium alloy composition.

15

20

25

30

- 9. The casting of any one of claims 6 to 8, wherein the primary particles exhibit a concentration ratio of magnesium to more dense alloy elements which decreases from the centre of the primary particles.
- 5 10. The casting of claim 9, wherein the ratio decreases in a decaying, fluctuating form.
 - 11. A process for producing a casting according to any one of claims 1 to 10, using a pressure casting machine having supply means for providing magnesium alloy in a molten state, a mould defining a die cavity of a shape required for the casting, and a flow path providing communication between the supply means and the die cavity, with part of the length of the flow path defining a controlled expansion port (hereinafter a "CEP") which increases in cross-sectional area in the direction of alloy flow along the flow path to the die cavity; wherein the process includes the steps of:
 - (a) causing molten alloy to flow from the supply means into the flow path such that, in its flow from the inlet end to the outlet end of the CEP, the alloy decreases in flow velocity whereby it is caused to undergo a change in state from the molten state to a semi-solid state;
 - (b) maintaining the alloy in the semi-solid state substantially throughout its flow into the die cavity; and
 - (c) causing solidification of alloy in the die cavity, and back along the flow path towards or into the CEP, at a sufficiently rapid solidification rate whereby the resultant casting has a microstructure characterised by fine primary particles in a matrix of secondary phase, with the primary particles of a form from the group consisting of rounded, spheroidal, degenerate dendritic and mixtures thereof, and substantially evenly distributed.
 - 12. The process of claim 11, wherein the decrease in flow velocity attained in step (a) and the solidification rate in step (c) are sufficient to provide primary particles, in the microstructure of the resultant casting, which are substantially less than 40 μ m in size.

13. The process of claim 11, wherein the decrease in flow velocity attained in step (a) and the solidification rate in step (c) are sufficient to provide primary particles, in the microstructure of the resultant casting, which are about 10 μ m or less.

5

10

14. The process of any one of claims 11 to 13, wherein the solidification rate in step (c) is such that solidification of alloy in the die cavity proceeds back into the CEP whereby alloy solidified in the CEP has a microstructure characterised by fine primary particles in a matrix of secondary phase, with the primary particles of a form from the group consisting of rounded, spheroidal, degenerate dendritic and mixtures thereof, and substantially evenly distributed, and further characterised in longitudinal sections of the CEP by striations on bands extending transversely of the CEP at a spacing between centres for successive like bands of the order of 40 µm.

15

15. The process of any one of claims 11 to 14, wherein the CEP is operable to cause the alloy to attain a flow velocity at the inlet end of the CEP which is in excess of 60 m/s and a flow velocity at the outlet end of the CEP which is from 50 to 80% of the flow velocity at the inlet end of the CEP.

20

16. The process of claim 15, wherein the CEP is operable to cause the alloy to attain a flow velocity at the inlet end of the CEP of from 140 to 165 m/s and a flow velocity at the outlet end of the CEP of from 70 to 130 m/s.

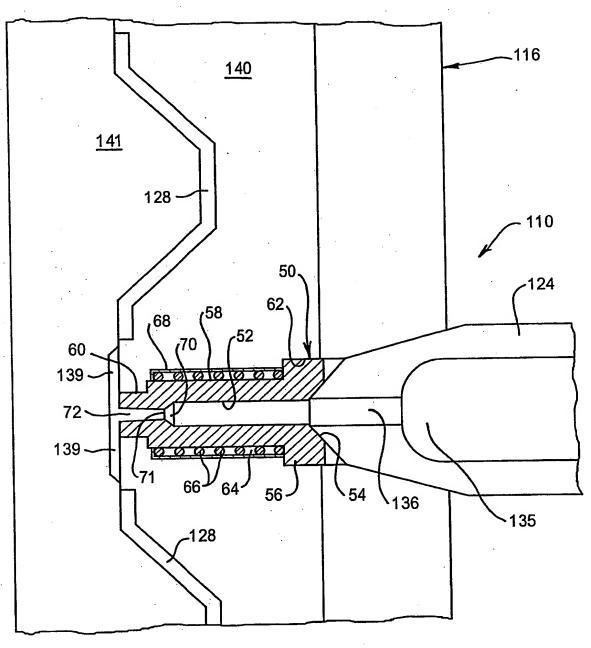
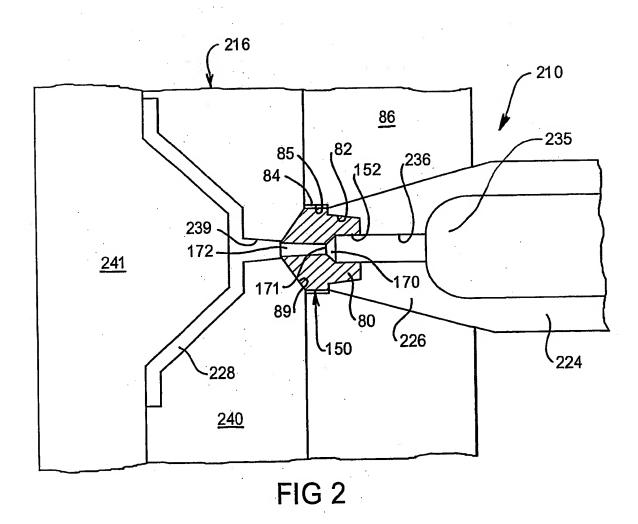


FIG 1



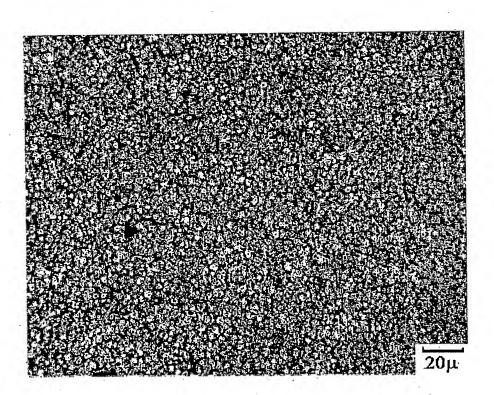


FIG 3

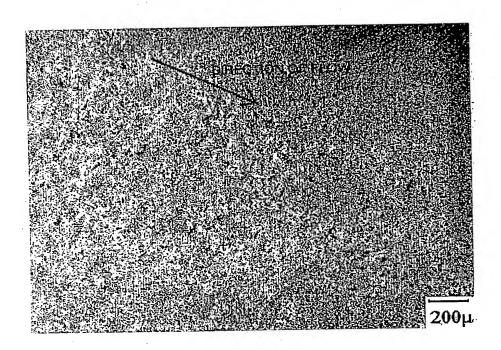


FIG 4

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU02/01140

| Α. | CLASSIFICATION OF SUBJECT MATTER | | | | | | |
|--|--|---|-----------------------|--|--|--|--|
| | B22D 17/20 | | | | | | |
| According to International Patent Classification (IPC) or to both national classification and IPC | | | | | | | |
| B. FIELDS SEARCHED | | | | | | | |
| Minimum documentation searched (classification system followed by classification symbols) IPC ⁷ as above and B22D 17/00, 17/02, 17/04, 17/06, 17/08, 17/10, 17/12 | | | | | | | |
| Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched | | | | | | | |
| Electronic data base consulted during the International search (name of data base and, where practicable, search terms used) Derwent WPAT: IPC ⁷ as above and B22D 17/00, 17/02, 17/04, 17/06, 17/08, 17/10, 17/12 and (expan+or taper+ or section+) | | | | | | | |
| C. DOCUMENTS CONSIDERED TO BE RELEVANT | | | | | | | |
| Category* | Citation of document, with indication, where appropriate, of the relevant passages | | Relevant to claim No. | | | | |
| Р, Х | WO 2002/16062 A1 (C. S. I. R. O.) 28 Feb Whole Document | oruary 2002 | 1 to 16 | | | | |
| Р, Х | WO 2002/30596 A1 (C. S. I. R. O.) 18 Apr Whole Document | ril 2002 | 1 to 16 | | | | |
| А | WO 1995/34393 A1 (Cornell Research Fo Whole Document | undation) 21 December 1995 | | | | | |
| | | f Box C X See patent family annu | | | | | |
| | Further documents are listed in the continuation o | f Box C X See patent family annu | | | | | |
| * Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document published prior to the international filing date "T" later document published after the international filing date and not in conflict with the application but cited to the principle or theory underlying the invention document of particular relevance; the claimed invention considered novel or cannot be considered to involve an inventive step when the document of particular relevance; the claimed invention considered to involve an inventive step when the document with one or more other such documents, such document member of the same patent family "X" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "B" document published after the international filing date and not in conflict with the application but cited to the principle or theory underlying the invention document of particular relevance; the claimed invention considered novel or cannot be considered to involve an inventive step when the document of particular relevance; the claimed invention considered to involve an inventive step when the document of particular relevance; the claimed invention considered to involve an inventive step when the document of particular relevance; the claimed invention considered to involve an inventive step when the document of particular relevance; the claimed invention considered to involve an inventive step when the document of particular relevance; the claimed invention considered to involve an inventive step when the do | | ntion cannot be ve an inventive ntion cannot be ocument is , such | | | | | |
| Date of the a | ed actual completion of the international search | Date of mailing of the international search repo | 27 SEP 2002 | | | | |
| • | nber 2002 | A. Harbard officer | | | | | |
| Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No. (02) 6285 3929 | | DAVID K. BELL Telephone No: (02) 6283 2309 | | | | | |

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU02/01140

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

| Patent Document Cited in Search Report | | Patent Family Member | | | | |
|---|-----------|----------------------|----------|----|-----------|-----------------|
| wo | 200230596 | AU | 20000763 | AU | 200195269 | |
| WO | 200216062 | AU | 20009678 | AU | 200181596 | |
| wo | 9534393 | EP | 765198 | US | 5501266 | |
| | | | | | | END OF ANNEX |

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

| Defects in the images include but are not limited to the items checked: | | | |
|---|--|--|--|
| ☐ BLACK BORDERS | | | |
| ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES | | | |
| FADED TEXT OR DRAWING | | | |
| BLURRED OR ILLEGIBLE TEXT OR DRAWING | | | |
| ☐ SKEWED/SLANTED IMAGES | | | |
| ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS | | | |
| ☐ GRAY SCALE DOCUMENTS | | | |
| ☐ LINES OR MARKS ON ORIGINAL DOCUMENT | | | |
| ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY | | | |
| | | | |

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.